CHAPTER

4

CHARACTERISATION OF EARLYWOOD AND LATEWOOD CTMP FIBRES AND THE EFFECT OF ENZYMATIC TREATMENT ON THEIR STABILITY

ABSTRACT

Softwood pulps are morphologically more variable than those of hardwoods, mostly in relation to thickness of cell-walls due to the presence of earlywood and latewood. In pulp, latewood fibres form weak inter-fibre bonds with bad conformation, while earlywood fibres form stronger bonds due to higher conformability achieved through refining. Sheet formation places stress on fibres and when inter-fibre bonds are broken, stress is relieved and fibres are allowed to return to their original shape. Thin-walled earlywood fibres conform better, but are less stable and move more when inter-fibre bonds are broken. This lack of stability can potentially be improved by treating the pulp with enzymes to improve conformation. The aim of the present study was to compare the stability of earlywood and latewood fibres and to study the impact of mannanase on fibre morphology, handsheet properties and stability. This study on pinewood chemi-thermo mechanical pulp showed that earlywood produced smoother handsheets with more strength, but less stability that resulted in an increase in roughness and puffing after wetting. Due to the bad conformation of the latewood fibres, less fibre movement was observed and the change in surface roughness was less obvious. Enzymatic treatment improved burst and tensile indices of latewood, possibly through improved fibrillation of the thick-walled fibres. Wetting of handsheets showed that fibre stability improved with mannanase, especially when earlywood fibres were treated. Handsheet smoothness and some of the strength properties were consequently retained after rewetting.

4.1. INTRODUCTION

Mechanical softwood pulps are characteristically more variable in terms of fibre morphology than hardwood pulps, with most of this variation occurring in the thickness of cell-walls (Reme *et al.*, 1999; Reme and Helle, 2001). This variation is mainly due to morphological differences between the earlywood (EW) and latewood (LW) fractions (Sjöström, 1993). Earlywood fibres have thin walls and large lumina (Sjöström, 1993), and their main function is to transport nutrients and water. Earlywood fibres absorb more energy during refining leading to better fibrillation, but are also easier to damage resulting in weakening of the cell-wall (Rudie *et al.*, 1994; Huang *et al.*, 2007). After refining, strong inter-fibre bonds form between the large contact areas provided through fibrillation and fibre collapse. The higher conformation of EW fibres results in smoother sheet surfaces with higher tensile index, but lower tearing resistance in comparison to sheets with more LW fibres (Paavilainen, 1992). In contrast, LW fibres are longer that EW fibres and have thicker cell-walls and smaller lumen volumes and serve more as mechanical support in trees (Biermann, 1996; Kure, 1999). However, LW fibres are mechanically more resistant, and fibrillate to a lower extent during refining (Huang *et al.*, 2007). These long, thick-walled fibres present in mechanical pulp collapse less, leading to smaller surface contact areas and thus weaker inter-fibre bonding (Forseth *et al.*, 1997). The poor inter-fibre bonding between the fibres can lead to rougher paper (Mohlin, 1989; Honksalo, 2004).

Calendering can improve surface smoothness possibly by forcing fibres closer together, encouraging greater fibre collapse and increasing inter-fibre contact and bonding. The greater conformation forced into fibres by calendering can, therefore, create higher strain among fibres. When moisture is increased, the bonds that maintain the position of these fibres in the paper web are broken, stress is relieved and fibres can return to their original shape (Forseth *et al.*, 1997). It was observed by Forseth and Helle (1996) that thin-walled fibres conform better during sheet formation, while thick-walled fibres mostly retain their original shape. Therefore, EW fibres revert to its tubular shape when rewetted such as during printing processes. However, in an earlier study, the effect of fibre morphology was investigated and it was shown that thin-walled fibres of spruce were less stable and moved more during rewetting handsheets (Strey *et al.*, 2009). That study also showed that fibre stability could be improved by treating mechanical pulp with enzymes such as mannanase (MAN) and endoglucanase (EG) before refining. The EG-treated pulp retained surface properties after rewetting, but not strength properties, whereas MAN led to retaining of both surface and strength properties.

Therefore, the aim of the present study was to compare the stability of EW (thin-walled) and LW (thick-walled) fibres and to study the impact of enzyme treatment on these fractions. Mannanase was selected for the treatment as it was the enzyme that gave the most promising results (Strey *et al*., 2009). The impact of the enzyme was evaluated on fibre characteristics and handsheet properties, before and after rewetting. This study was conducted on pinewood, since pine contains a high ratio of LW to EW when compared to either spruce or fir (Lindström *et al*., 1977; Varhimo and Tuovinen, 1999). Pine pulp thus produced an ideal furnish to test the potential of MAN to improve pulp stability.

4.2. MATERIALS AND METHODS

4.2.1. Wood sampling and pulping

A *Pinus patula* log (approximately 12 years old) was sawn into disks of about 2 cm thick. Each disk was then sawn into strips (3 cm wide) and segmented with a chisel to produce EW and LW chips. The EW and LW fractions were each separated into six 50 g samples to be pulped and treated separately. Each sample was pulped in cooking liquor in its own container, but pulping was done simultaneously in one digester. The pulping conditions and fibre separation in a refiner simulated a chemi-thermo-mechanical pulping (CTMP) process. A charge of 6.6% sodium bisulphite with liquid to wood ratio of 2.5 : 1, a ramp-up time of 90 min and a reaction time of 60 min at 165°C were used. After cooking, fibres were separated using a three-stage process on a Sprout-Waldron refiner. On the first pass through the refiner a 1.1 mm gap was used followed by two passes with the gap set at 0.4 mm. Fibre characteristics and strength properties were determined to confirm effective separation of EW and LW.

4.2.2. Enzyme treatment

A sample of 1ℓ of pulp at a 3.5% consistency in tap water was prepared from each replicated fraction, in order to assess the impact of MAN on each of the replications of the EW and LW fractions. The pH was adjusted to 7.0 using H_2SO_4 (0.1M). Mannanase NZ 51023 (MAN) from Novozymes, Denmark was applied at a dosage of 360 U g^{-1} pulp and water was used as a control. The treatment was incubated at 60°C for 2 h with manual shaking every five to 10 min and was based on the conditions existing in the mill. After treatment, each sample was beaten at 3000 revolutions using a PFI mill. The samples were then disintegrated at 1500 revolutions using a MK IIIC disintegrator (Messmer Instruments Limited, UK), where after it was diluted to approximately 6 ℓ with tap water and left overnight to reduce latency.

4.2.3. Fibre characterisation and handsheet properties

Fibre characteristics for both EW and LW samples after fibre separation and after beating were determined using a MorFi LB-01 Fibre Quality Analyser (TECHPAP, France). These fibre characteristics included cell-wall thickness (CWT), fibre length, fibre width, coarseness, density, curl, kinked fibres, broken ends and the proportion of fines.

Ten handsheets with a basis weight of 60 g m^{-2} were prepared for each sample according to the Rapid-Köthen method (ISO 5269/2). Each set of handsheets was divided into two groups. One group was kept dry and conditioned overnight at a relative humidity of 50% and 23°C. The second group was rewetted by briefly dipping handsheets into water before conditioning to a constant weight under the conditions described above. The surface roughness, tensile and burst indices of handsheets were determined using ISO 8791-2 (1990), ISO 1924 (2005) and ISO 2758 (2001), respectively.

4.2.4. SEM examination

Three samples (3 x 10 mm) were randomly cut to be representative of the surface of the handsheets and embedded in Quetol 651 (Van der Merwe and Coetzee, 1992). A microtome was used to obtain transverse sections through the middle of the sample, and the sectioned face immersed in a saturated solution of sodium methoxide for approximately 40 min to etch the resin away from the fibres and thus expose a clean transverse section of the handsheet (Iwadare *et al.*, 1990; Chapter 2). Samples were briefly rinsed in methanol, air dried, mounted on stubs with double-sided carbon tape with etched surfaces uppermost, and rendered conductive in the vapour of a 0.5% (w/v) RuO_4 solution (Van der Merwe and Peacock, 1999).

A SEM (JSM 840, JEOL, Tokyo, Japan) at 5 kV and a working distance of 10 mm was used to record five electron micrographs per replicated treatment. Transverse dimensions of the fibre and lumen were measured by using ImageTool software (University of Texas, www.http://ddsdx.uthscs.edu/) and calculations of the ratio of lumen area to fibre area (LA:FA) were done as described previously to quantity puffing (Chapter 2).

4.2.5. Experimental design and statistical analysis

A completely randomised experimental design with three factors (wood fraction, enzyme treatment and handsheet wetting) was used to compare fibre morphology, handsheet properties and fibre stability for each treatment. The whole data set was initially subjected to analysis of variance, but because all interactions were significant, the impact of each factor was evaluated separately by doing one-way ANOVA and means tested for significant differences (Q-value) with Tukey's multiple-range test at a 95% confidence level.

4.3. RESULTS AND DISCUSSION

4.3.1. Pulp characteristics

Earlywood consists of thin-walled fibres, while LW fibres are longer with thicker walls (Sjöström, 1993). In the present study, the significant difference in CWT between fractions confirmed that EW and LW had been successfully separated before pulping (Fig. 4-1, Table 4-1). Fibres in the EW fraction had a CWT of approximately 3 µm, whereas that of the LW fibres was 6 µm (Fig. 4-1). The fibres in the LW fraction were also significantly longer than the EW fibres, however, fibre characteristics such as width, density and curl did not differ significantly between the two fibre fractions (Table 4-1).

Coarseness was higher in LW fractions (Table 4-1) reflecting the longer and thicker fibres as described by Sjöström (1993). More EW fibres were kinked in comparison to LW fibres after refining (Table 4-1), possibly because EW fibres absorbed more energy, resulting in structural changes during refining (Huang *et al*., 2007). In LW fibres, broken ends increased (Table 4-1) suggesting that fibres broke rather than kinked due to their stiff, unyielding structure as described by Forseth *et al*. (1997). The amount of fines was significantly higher in the LW fraction and accounted for the reduction in the fibre length that was observed (Table 4-1). Corson and Ekstam (1994) also observed that LW fibres were more often broken down into fines than EW fibres.

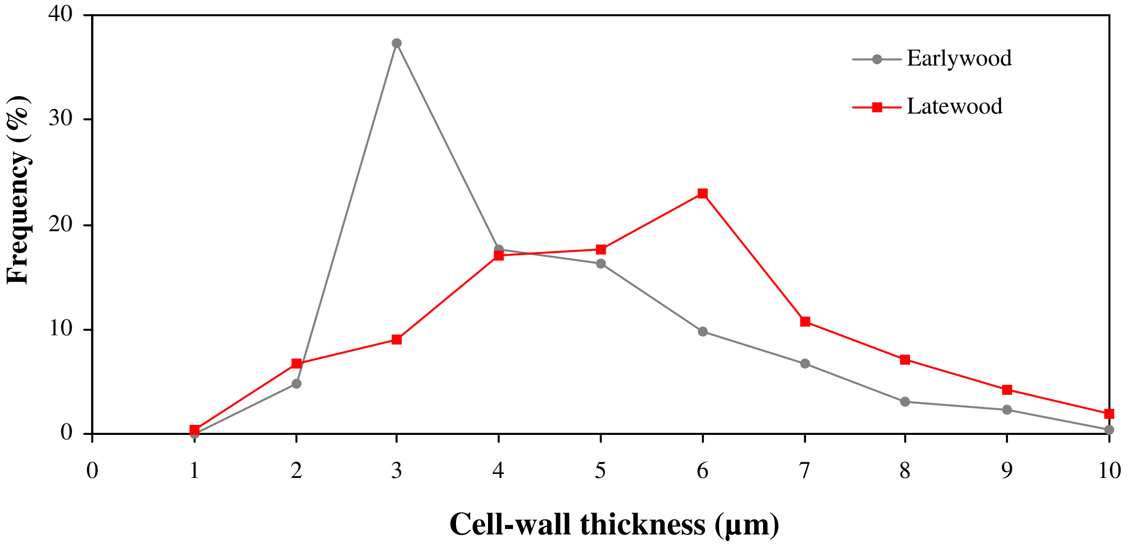

Figure 4 - 1: *Distribution of cell-wall thickness in earlywood and latewood fibres in laboratory CTMP.*

Table 4 - 1: *Fibre characteristics of pine earlywood and latewood after beating with a PFI mill as determined with the MorFi fibre analyser.*

Fibre characteristics	**Earlywood**	**Latewood**
Average cell-wall thickness (µm)	4.180^{a}	5.518^{b}
Fibre length: weighted in length (µm)	1313^{a}	1431^{b}
Fibre width (µm)	40.13^{a}	38.43^{a}
Fibre coarseness (mg g^{-1})	0.65^{a}	0.94^{b}
Fibre density (million g^{-1})	1.80^{a}	1.35^{a}
Curl (%)	9.00^{a}	8.90^{a}
Kinked fibres (%)	26.23^{a}	24.97^{b}
Fines (% length)	72.13^{a}	78.80^{b}
Broken ends (%)	51.67^{a}	56.18^{b}

a b: Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$, Tukey's Multiple-Range test).

4.3.2. Influence of earlywood and latewood fractions on handsheet properties

The thick-walled LW fibres yielded handsheets with higher surface roughness when compared to EW fibres (Table 4-2), possibly reflecting the presence of coarse fibres that did not collapse or conform well. Fibres that do not have a high degree of conformation result in smaller contact areas between fibres and weaker inter-fibre bonding. This is supported by the fact that burst and tensile indices were lower for LW handsheets than for EW sheets (Table 4-2).

Table 4 - 2: *Properties of handsheets from pine earlywood and latewood pulps after beating with a PFI mill.*

Handsheet properties	Earlywood	Latewood
Roughness (ml min^{-1})	1423^{a}	3648^{b}
Tensile index (mN m^{2} g^{-1})	18.59^{a}	16.06^{b}
Burst index (kPa m^{2} g^{-1})	0.675^{a}	0.421^{b}

a b: Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$, Tukey's Multiple-Range test).

4.3.3. Influence of mannanase on fibre characteristics

Treatment with MAN did not yield significant changes in most of the fibre characteristics of EW, but the coarseness of EW fibres was reduced significantly, possibly reflecting improved flexibility (Table 4-3). On the LW fraction, MAN treatment did not result in any significant changes in the fibre characteristics (Table 4-4). These results confirm previous results where enzymes did not induce any significant changes in the morphological characteristics of spruce fibres (Strey *et al*., 2009).

Table 4 - 3: *Fibre characteristics of earlywood fibres before and after mannanase (MAN) treatment and beating with a PFI mill.*

Fibre characteristics	Control	MAN
Fibre length: weighted in length (µm)	1316^{a}	1322^{a}
Fibre width (µm)	40.13^{a}	39.90^{a}
Fibre density (million g^{-1})	2.13^{a}	2.48^{a}
Curled fibres (%)	9.00^{a}	8.83^{a}
Kinked fibres (%)	23.23^{a}	23.17^{a}
Fibre coarseness (mg g^{-1})	0.71^{a}	0.49^{b}
Fines: length (%)	72.13^{a}	72.63^{a}

a b: Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$), Tukey's multiple-range test.

Table 4 - 4: *Fibre characteristics of latewood fibres before and after mannanase (MAN) treatment and beating with a PFI mill.*

Fibre characteristics	Control	MAN
Fibre length: weighted in length (µm)	1414	1445
Fibre width (µm)	38.43	37.93
Fibre density (million g^{-1})	1.34	1.79
Curled fibres (%)	8.30	8.87
Kinked fibres (%)	19.97	20.97
Fibre coarseness (mg g^{-1})	0.94	0.70
Fines: length (%)	78.80	77.83

No significant difference at $p \leq 0.05$

4.3.4. Influence of mannanase on handsheet properties

In EW pulp, the enzyme treatment resulted in the formation of smoother handsheet surfaces and also increased burst strength. However, no significant changes in the tensile strength were observed (Table 4-5). Mannanase improved properties of EW pulp (Table 4-5), confirming the findings from Strey *et al.* (2009), where MAN reduced roughness and increased tensile and burst indices. Since both smoothness and burst were improved, it is possible that enzyme treatment led to higher fibre collapsibility and cell-wall fibrillation.

Table 4 - 5: *The influence of mannanase (MAN) treatment on dry handsheet properties from earlywood and latewood.*

Wood fraction	Handsheet properties (dry)	Control	MAN
Earlywood	Roughness (ml min^{-1})	1423^a	1277^b
	Tensile index (mN m^2 g^{-1})	19.52^a	18.37^a
	Burst index (kPa m^2 g^{-1})	0.675^a	0.717^b
Latewood	Roughness (ml min^{-1})	3648^a	3746^a
	Tensile index (mN m^2 g^{-1})	13.21^a	13.78^b
	Burst index (kPa m^2 g^{-1})	0.419^a	0.470^b

a b: Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$), Tukey's multiple-range test.

It is difficult to modify fibres with thicker cell-walls (such as LW fibres) through refining (Huang *et al*., 2007) and, therefore, only a small effect can be obtained on the fibre morphology and handsheet properties (Kure, 1999). However, in the present study MAN treatment followed by refining increased the tensile and burst indices of LW-containing handsheets significantly (Table 4-5), probably due to the better fibrillation of enzyme-modified cell-walls. Beating of these thick-walled LW fibres after enzyme treatment did not have a significant impact on the surface properties of handsheets (Table 4-5), suggesting that the enzymes did not improve collapsibility and conformability to a larger extent.

4.3.5. Stability of earlywood fibres

When handsheets from untreated softwood pulp were rewetted, surface roughness increased and strength properties were reduced, possibly as result of movement of unstable fibres (Skowronski and Lepoutre, 1985; Strey *et al*., 2009). In the present study, handsheets from untreated EW pulp also showed a significant increase in roughness when rewetted (Table 4-6), supporting the earlier suggestion that fibres reverted to their original shape and this disrupted the paper web. As before with spruce, treatment of pine fibres with MAN controlled this fibre movement and surface roughness could be maintained within 99% of the dry control (Table 4-6).

Table 4 - 6: *The influence of treatment with mannanase (MAN) and rewetting on pulp properties of handsheets from earlywood pulp.*

Handsheet properties	Control (dry)	Control (rewetted)	MAN (rewetted)
Roughness (ml min^{-1})	1423^a	1562^b	1405^a
Tensile index (mN m^2 g^{-1})	19.52^a	16.80^a	17.03^a
Burst index (kPa m^2 g^{-1})	0.675^a	0.607^a	0.706^b

a b: Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$), Tukey's multiple-range test.

In previous investigations on spruce pulp (Strey *et al*., 2009), the tensile as well as the burst indices of untreated pulp decreased after rewetting, but in the present study, tensile and burst indices were not significantly different before and after rewetting (Table 4-6). This lack of puffing due to rewetting may be typical of the thicker-walled pine fibres used in the present study. The different levels of reaction to water may also be explained by the structure and chemical differences between these species (Sjöstrom, 1993). When these EW fibres were treated with MAN, a significant increase in burst index was observed before rewetting (Table 4-5) and this improvement was retained even after rewetting (Table 4-6). Therefore, MAN has also the potential to modify cell-walls of EW fibres, increase collapsibility and strengthen inter-fibre bonds. However, this effect was not evident for tensile strength (Table 4-6).

4.3.6. Stability of latewood fibres

After rewetting handsheets made from LW, no significant changes were observed in surface roughness (Table 4-7). This is not surprising, as it was previously demonstrated that thick-walled fibres (such as those found in LW) did not collapse in the sheet, and thus retained their rigid structure (Strey *et al.*, 2009). The fact that the roughness of handsheets from LW (Table 4-7) fibres in this study was more than double the roughness of that measured in EW handsheets (Table 4-6) confirms the poor collapsibility before and after rewetting.

The tensile index of the untreated handsheets was significantly reduced after rewetting (Table 4-7). Bonding was improved with MAN treatment before rewetting (Table 4-5) but, in contrast to EW, the stability of these bonds was not maintained after rewetting (Table 4-7). The reduction in tensile strength of LW may reflect less fibre fibrillation during refining, causing weaker inter-fibre bonding that was easily broken in the presence of water. The burst index, on the other hand, was not significantly influenced for the control or enzyme-treated handsheets when subjected to rewetting.

Table 4 - 7: *The influence of mannanase (MAN) treatment rewetting on pulp properties of handsheets, from latewood pulp.*

Handsheet properties	Control (dry)	Control (rewetted)	MAN (rewetted)
Roughness (ml min^{-1})	3648^a	4236^a	3918^a
Tensile index (mN m^2 g^{-1})	13.21^a	10.09^b	11.23^b
Burst index (kPa m^2 g^{-1})	0.419^a	0.410^a	0.433^a

a b: Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$), Tukey's multiple-range test.

4.3.7. SEM examination

Calculating the ratio of lumen area to fibre area (LA:FA) appeared to be a suitable parameter to evaluate the effect of rewetting on fibre stability, as demonstrated previously (Strey *et al.*, 2009). The stability of EW and LW was, therefore, quantified by calculating the LA:FA ratio of dry and rewetted handsheets. The LA:FA ratio in untreated EW fibres increased significantly (by 35%) after rewetting, indicating that the lumen volume increased as fibres puffed (Table 4-8). However, in LW handsheets the LA:FA increased by 10%, but this change was not significant when comparing dry and rewetted controls. Enzymatic treatment allowed fibre shape to be maintained for both EW and LW fibres when compared to the dry control samples. Fibre puffing occurred in untreated EW fibres, and it was possible to reduce this effect with MAN treatment (Table 4-8), leading to a smoother paper with higher strength.

Table 4 - 8: *Ratio of lumen area (LA) to fibre area (FA) as a reflection of the degree of puffing before and after rewetting for untreated and treated earlywood and latewood fibres.*

Wood fraction	Control (dry)	Control (rewetted)	MAN (rewetted)
Earlywood (LA:FA)	0.212^a	0.326^b	0.140^a
Latewood (LA:FA)	0.141^a	0.157^a	0.133^a

a b: Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$), Tukey's multiple-range test.

4.4. CONCLUSIONS

Softwood is a heterogeneous material containing EW and LW fibres, with different fibre characteristics that influence the handsheet properties (Vomhoff and Grundström, 2003). In the present study the EW fraction of pine CTMP contained shorter fibres with thinner cell-walls and less coarse structure and produced handsheets with a much smoother surface and higher strength when compared to the LW fibres. The surface smoothness obtained with EW fibres reflected higher collapsibility and better conformation, while improved strength revealed better fibrillation. However, EW fibres were unstable, which was reflected in an increase in roughness and puffing (LA:FA ratio) when subjected to moisture. However, the strength properties were retained possibly due to good conformation.

Instability of LW fibres was seen only in the reduction of tensile strength and possibly reflected more water penetration into the open structure of badly-conformed sheets. Due to the bad conformation of these fibres, less fibre movement was observed and the change in surface roughness was less obvious. In agreement with work reported by Hattula and Niemi (1988), CWT appeared to be the dominant factor in determination of fibre instability.

Enzymatic treatment improved burst and tensile strengths of LW, possibly through improved fibrillation of the thick-walled fibres during beating with a PFI mill. Fibre stability was improved with MAN treatment especially when EW fibres were treated. Handsheet smoothness and some of the strength properties were consequently retained after rewetting. Increased fibre stability after MAN treatment was confirmed through SEM and image analysis of fibres in cross-sections.

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CHAPTER
5

THE IMPACT OF ENZYME TREATMENT ON THE FIBRE STABILITY OF POPLAR CTMP

ABSTRACT

Blends of softwood and hardwood fibres are generally used in mechanical paper grades to obtain the required smoothness and strength properties. Hardwood fibres are in general thinner and collapse easier, and are included in the mixture to contribute to surface smoothness. Moisture can lead to deterioration of both smoothness and strength, due to the instability of fibres. In the present study, when handsheets from hardwood pulp were exposed to water, surface roughness was not increased. However, strength properties (tensile and burst) were reduced. It was possible to retain these strength properties by treating fibres with mannanase while most of the strength properties deteriorated after endoglucanase treatment. Since wetting did not influence pulp properties to the same extent as observed previously in softwood, it was concluded that hardwood fibres were more stable. This can possibly be explained by the difference in fibre length and coarseness and chemical composition of the cell-wall. The present study confirmed that it is possible to use a mannanase treatment on a mixed furnish containing softwood and hardwood without any detrimental effects.

5.1. INTRODUCTION

Printing performance is becoming increasingly important to all paper grades containing mechanical pulp. These quality aspects include adequate strength and smooth surface properties after coating and printing. However, due to differences in the fibre morphology (e.g. fibre length, width and cell-wall thickness) the pulp quality can vary (Dinwoodie, 1965; Horn, 1974, 1978). Softwood pulps typically contain long fibres (2 to 4 mm), with a width of 0.02 to 0.04 mm and cell-wall thicknesses (CWT) that vary between 2 and 4 µm in earlywood and 4 to 8 µm in latewood. In contrast, hardwood pulps contain shorter fibres that are only 1.1 to 1.2 mm in length, 0.014 to 0.04 mm wide with an average CWT of 3 to 4.4 µm (Sjöström, 1993).

It is generally accepted that paper with desirable strength properties must include long fibres such as those of softwood pulps (Dadswell and Wardrop, 1959; Barefoot *et al*., 1964; Horn, 1978). However, the increased fibre length, width and CWT associated with softwood are accompanied by a corresponding decrease in fibre collapsibility (Forseth and Helle, 1996; Forseth *et al*., 1997), which can lead to an increase in surface roughness. To overcome this, hardwoods with typically thinner cell-walls and higher flexibility are used in blends with softwood to obtain the required surface smoothness (Sjöström, 1993). Additionally, the modification of softwood fibres through enzyme treatment can also be used successfully to produce more stable, smoother and stronger paper (Strey *et al*., 2009; Chapter 4). Enzymes such as endoglucanase (EG) and mannanase (MAN) used on their own or in combination reduced surface roughness, while MAN also contributed to better strength properties (Strey *et al*., 2009; Chapter 4). However, since most of the mechanical pulps are blends of softwood and hardwood fibres, it was necessary to investigate the effect of these two enzymes on the hardwood fibres, and this forms the subject of this chapter.

Although hardwoods have more flexible fibres with thinner cell-walls, they contain approximately the same amount of cellulose as softwood (Sjöström, 1993). However, the composition and structure of the hemicelluloses in hardwoods differ from that of softwoods. The main component of hardwoods is xylan (15 to 30%), with only 2 to 5% glucomannan, compared to the 25 to 30% glucomannan, which is the main component in softwood.

Accordingly, the enzymes tested on softwood (Strey *et al*., 2009; Chapter 4) may not have the desired impact on hardwood fibres or may even have negative effects when mixed furnishes are treated. The aim of the present study was, therefore, to determine the effect of EG and MAN on fibre characteristics and pulp properties of hardwood fibres. Conducting this study on a single hardwood species reduces the potential complexity of the response from a mixed furnish. The influence of the enzymes was assessed by monitoring fibre characteristics, handsheet strength and stability of poplar fibres during rewetting.

5.2. MATERIALS AND METHODS

5.2.1. Pulp preparation

Poplar chemi-thermo-mechanical pulp (CTMP) was produced in the laboratory at the Sappi Technology Centre (STC) at Pretoria under milder conditions than those used for the pulping of spruce (Strey *et al*., 2009). These conditions were chosen to compensate for the thinner cell-walls of hardwood fibres, which make these more reactive towards pulping conditions and, therefore, requiring milder processing. The wood was pulped by cooking chips using a ramp-up time to 165°C of 60 min, followed by 60 min at 165°C and a 1% sodium-bisulphite charge and a liquor to wood ratio of 2.5 : 1. After pulping, two-stage refining was done to separate and fibrillate fibres. The first-stage refining was performed with a Sprout Bauer refiner at the CSIR (Forestry and Forest Products Research Centre, Durban). On the first pass through this refiner a 1.1 mm gap was used followed by two passes with the gap set at 0.4 mm. The second refining stage was performed with a low-consistency pilot refiner at the STC. During secondary refining, milder conditions that involved low intensity plates and a programme for hardwoods were used.

5.2.2. Enzyme treatment

Two enzymes were used: mannanase NZ 51023 (MAN) and endoglucanase Novozyme 476 (EG), both from Novozymes, Denmark. Combinations of these two enzymes were found to be detrimental to the strength properties of handsheets made from spruce fibres possibly due to a reduction in the fibre strength (Strey *et al*., 2009). Therefore, the enzyme combination of MAN and EG was not used in the present study. Pulp samples were prepared at a consistency of 3% and treated separately with MAN and EG for 2 h by following the same method as previously (Strey *et al*, 2009).

5.2.3. Freeness and fibre characterisation

The freeness (Canadian Standard Freeness) of each pulp sample was determined with a DFR 04 analyser (Mütek, Germany), followed by evaluation of the fibre characteristics with a MorFi LB-01 Fibre Quality Analyser (TECHPAP, France).

5.2.4. Handsheet properties

Handsheets with a basis weight of 60 g m^{-2} were prepared from each sample according to the ISO 5269/2 Rapid-Köthen method. The handsheets were divided into two groups, where the first group was kept dry and the second group was rewetted by dipping into water and placing them out to dry and condition overnight. Roughness, porosity, burst and tensile indices were determined for all the samples using the appropriate ISO methods (Strey *et al*., 2009). The stability of pulp was determined by comparing the properties of the rewetted handsheets with that of the dry control as described previously (Strey *et al*., 2009).

5.2.5. SEM examination

Three random samples were cut from each handsheet and embedded in Quetol 651 (Van der Merwe and Coetzee, 1992). A microtome was used to make sections in the middle of the sample and the sectioned face was then immersed in a saturated solution of sodium metoxide for approximately 40 min to etch the resin away from the fibres and expose a clean transverse section of the handsheet (Iwadare *et al*., 1990). Samples were air dried, mounted with double-sided carbon tape on stubs after which they were rendered conductive in the vapour of a 0.5% RuO_4 solution (Van der Merwe and Peacock, 1999). Scanning electron microscopy (SEM) (JSM 840, JEOL, Tokyo, Japan) was used to examine the samples at 5 kV and a working distance of 10 mm. Ten representative electron micrographs were recorded for each sample and analysed with ImageTool software (University of Texas, San Antonio, www.http://ddsdx.uthscs.edu). Transverse dimensions of the fibre-walls and lumina were measured and the ratio of the lumen area (LA) to fibre area (FA) was calculated as described previously (Strey *et al*., 2009; Chapter 2). This ratio quantifies the collapsibility of fibres and a change in this ratio reflects unstable fibres that undergo a movement referred to as puffing.

The median of the observed CWT was used to separate the data from thin- and thick-walled fibres for separate statistical analysis of fibre populations that behave differently in paper sheets (Strey *et al*., 2009).

5.2.6. Experimental design and statistical analysis

A completely randomised experimental design was used to test changes in characteristics of enzyme treated pulp before and after rewetting. Each treatment was replicated three times. The data were subjected to a one-way analysis of variance and means were tested for significant differences (Q-value) with Tukey's multiple-range test at a 95% confidence level.

5.3. RESULTS AND DISCUSSION

5.3.1. Freeness and fibre characterisation

Treating of poplar CTMP with MAN did not cause a significant difference in fibre length in comparison to the control after beating, but a significant reduction in fibre length was observed after treatment with EG (Table 5-1). This reduction in length could indicate that the cell-walls of poplar fibres are more responsive towards EG-treatment than MAN-treatment, reflecting the presence of amorphous cellulose. Endoglucanase treatment caused weak areas in the cell-wall structure making fibres more fragile and prone to breaking during beating. None of the enzymatic treatments resulted in a change in the fibre width. The coarseness and fibre density were reduced by approximately 50% following MAN treatment, but EG did not change any of these characteristics. The reduction in coarseness and density possibly reflect greater swelling and fibrillation of the MAN-treated fibres, but also some loss of cell-wall material as fines that reflect the increase in the percentage fines (Table 5-1). Both enzymes (MAN and EG) reduced curl, possibly as result of relaxation of the fibre structure. Although the percentage kink did not increase after enzymatic treatment, an increase was observed in the percentage broken ends after MAN treatment, but EG did not have any significant effect (Table 5-1).

Table 5 - 1: *Fibre characteristics of untreated (control) poplar fibres and fibres treated with mannanase (MAN) and endoglucanase (EG).*

Fibre characteristics	**Control**	**MAN**	**EG**
Fibre length weighted in length (µm)	661^{a}	547^{ab}	534^{b}
Fibre width (µm)	31.94^{ab}	32.50^{a}	31.43^{b}
Fibre coarseness (mg g^{-1})	0.64^{a}	0.315^{b}	0.62^{a}
Fibre density (g $million^{-1}$)	0.29^{a}	0.15^{b}	0.27^{a}

Curl (%)	9.38^a	7.50^b	7.13^c
Kinked fibres (%)	19.50^a	17.27^b	16.87^b
Fines (% length)	79.66^b	83.20^a	84.63^a
Fines (% area)	26.47^b	37.90^a	39.41^a
Broken ends (%)	47.93^b	50.42^a	48.38^b

a b c: Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$, Tukey's test).

5.3.2. Influence of enzymes on handsheet properties before rewetting

Roughness was not changed with either MAN or EG treatments and tensile and burst indices of the handsheets were not affected by MAN treatment (Table 5-2). However, EG reduced the tensile and burst indices significantly, possibly as a result of the degradation of cellulose in the cell-wall which led to loss of fibrillation and lower bonding strength after beating. The reduction in fibre length after treatment with EG (Table 5-1) also likely contributed to the poor strength properties (Table 5-2).

Table 5 - 2: *The influence of different enzyme treatments on dry handsheet properties.*

Handsheet properties (dry)	**Control**	**MAN**	**EG**
Roughness (ml min^{-1})	2798^a	2597^a	2570^a
Tensile index (mN m² g^{-1})	37.71^a	32.53^a	25.72^b
Burst index (kPa m² g^{-1})	1.706^a	1.679^a	1.549^b

a b : Values in each row followed by the same letter do not differ significantly ($p \leq 0.05$, Tukey's test).

5.3.3. Response of handsheet properties to rewetting

A reduction in surface smoothness and strength properties in handsheets after rewetting can be used as an indication of unstable fibres (Strey *et al.*, 2009; Chapter 4). In contrast to spruce fibres, roughness was not influenced by rewetting either before or after enzyme treatment (Fig. 5-1). The unaltered surface properties may be due to hardwood fibres being less coarse and, therefore, more stable. However, in softwood, roughness increased after rewetting, but enzymatic treatment preserved the surface smoothness after rewetting (Strey *et al.*, 2009; Chapter 4).

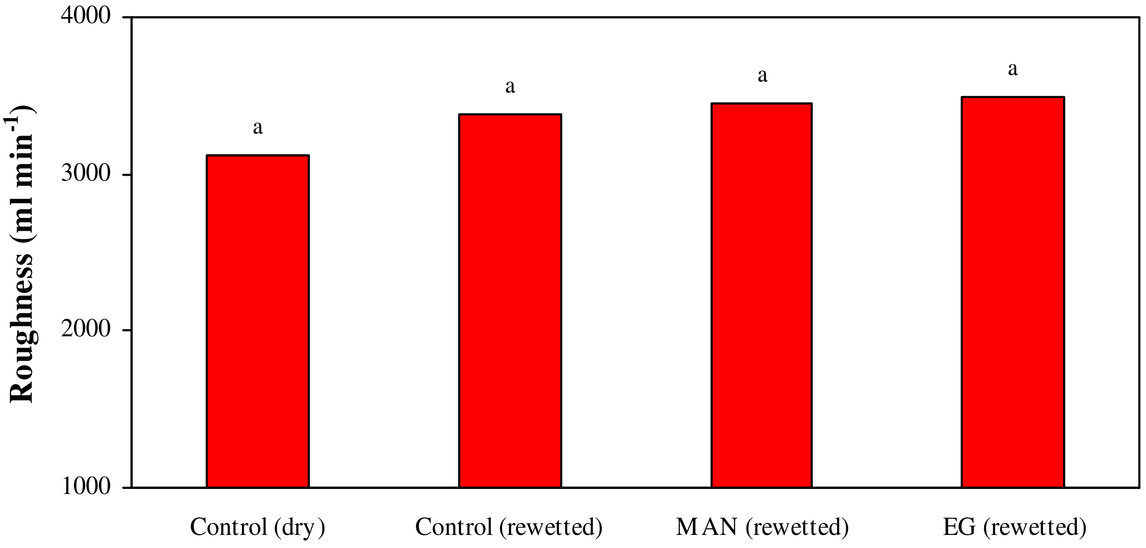

Figure 5 - 1: *The influence of rewetting on the roughness of handsheets from untreated and enzyme treated pulp ($p \leq 0.05$, Tukey's multiple-range test).*

A significant decrease in tensile and burst indices was found after rewetting the control handsheets, but strength was retained when MAN-treated handsheets were rewetted (Fig. 5-2 and Fig. 5-3). This retention of tensile strength was not observed after treatment with EG, and lower strength was measured compared to the control after handsheets were rewetted. Similar trends were observed on softwood pulp (Strey *et al*., 2009), indicating that EG damages the fibres, causing a reduction in strength similar to strength loss described by Mohlin and Petersson (2002) when using a multi-component cellulase.

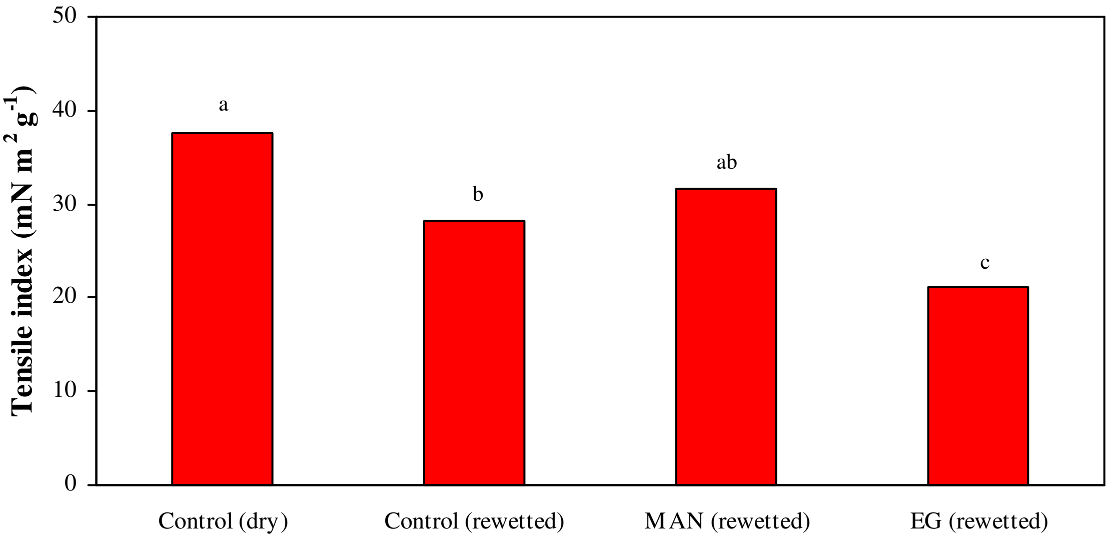

Figure 5 - 2: *The Influence of rewetting on the tensile index of handsheets from untreated and enzyme treated pulp ($p \leq 0.05$, Tukey's multiple-range test).*

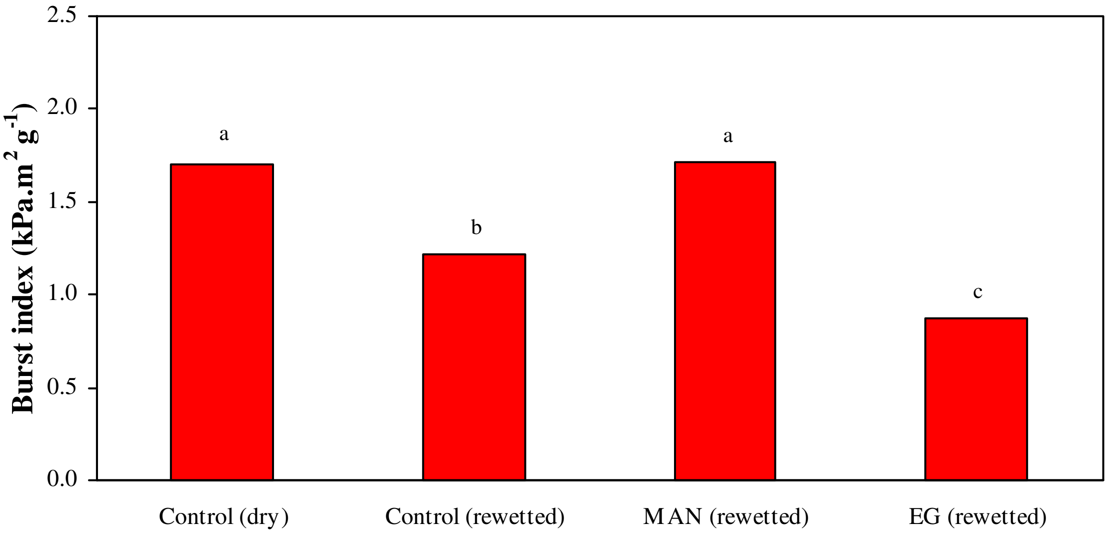

Figure 5 - 3: *The influence of rewetting on the burst index of handsheets from untreated and enzyme treated pulp ($p \leq 0.05$, Tukey's multiple-range test).*

5.3.4. SEM examination

Previously (Strey *et al.*, 2009) separated data for thin and thick-walled spruce fibres revealed the positive effects of enzymes on puffing. A similar approach was followed when large variations in the data for poplar fibres were observed. The data for thick-walled fibres (cell-walls thicker than a median of 2.04 µm) and the thin-walled (< 2.04 µm) fibres were then analysed separately. Rewetting did not have any significant effect on the LA:FA ratio of fibres with either thin- or thick-walls (Figs. 5-4 and 5-5). When these fractions were subjected to enzyme treatment, a slight decrease in LA:FA was observed, but it was not significant for either of the enzymes. Hardwood fibres, therefore, appeared to be more stable than softwood, possibly due to their lower coarseness (Sjöström, 1993).

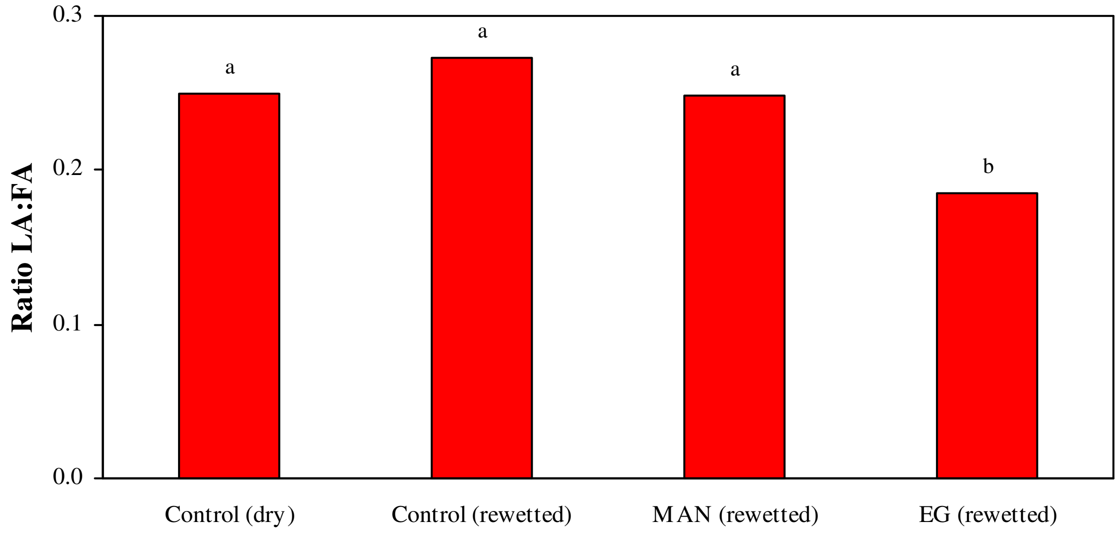

Figure 5 - 4: *The influence of rewetting, mannanase (MAN) and endoglucanase (EG) on puffing of thin-walled fibres ($p \leq 0.05$, Tukey's multiple-range test).*

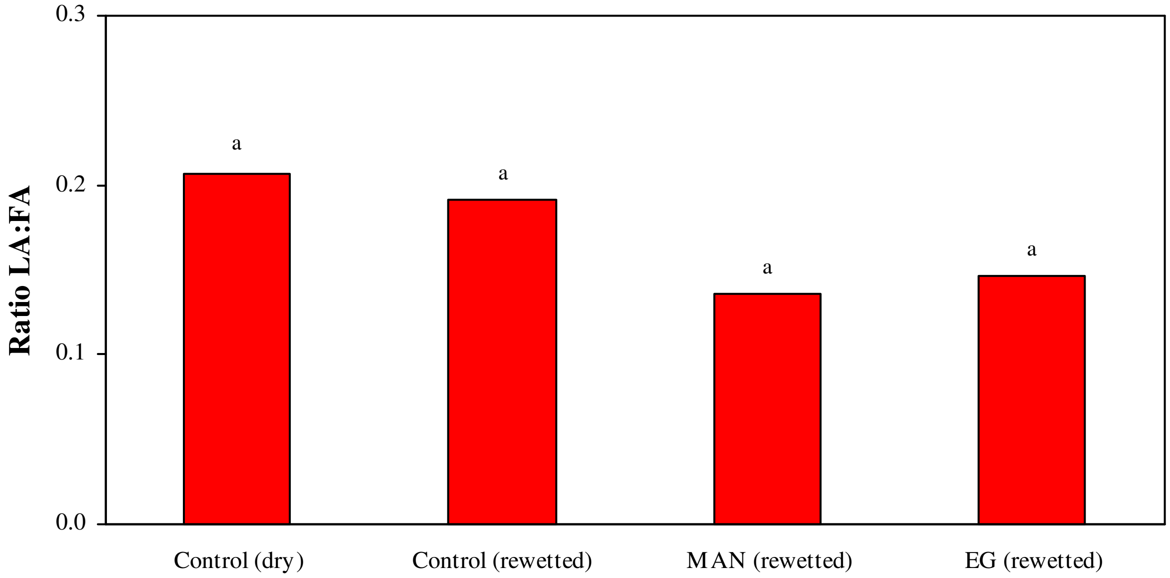

Figure 5 - 5: *The influence of rewetting, mannanase (MAN) and endoglucanase (EG) on puffing of thick-walled fibres ($p \leq 0.05$, Tukey's multiple-range test).*

5.4. CONCLUSIONS

The effect of enzymatic treatment on a mixed furnish (containing softwood and hardwood) has not been described previously. However, a study of single-species pulp can reduce the complexity when the response of fibres to rewetting as well as enzymatic modification is investigated. The response of fibres can be influenced by the differences in fibre characteristics and biochemical composition of the cell-wall between different species (Sjöström, 1993).

Hardwood fibres are often included in a mixed furnish to increase the paper smoothness (Varhimo and Tuovinen, 1999), due to their thin cell-walls and high degree of collapsibility. In the present study it was observed that rewetting and enzyme treatment did not influence the surface roughness of handsheets from hardwood. This study confirmed the role of hardwood to improve smoothness. Paper strength, on the other hand, was reduced after rewetting but when treated with mannanase these strength properties were maintained, but not after EG treatment, possibly due to fibre damage by EG (Mohlin and Petersson, 2002).

When MAN is applied to mixed pulps, the effect on hardwood fibres is not expected to be detrimental, and most of the paper properties (smoothness and strength) can be retained after rewetting. However, the presence of EG in a mixed furnish might degrade the strength properties of the hardwood fraction of the pulp.

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CHAPTER

6

FIBRE STABILITY IN COMMERCIAL PRODUCED PAPER SAMPLES FROM DIFFERENT PRODUCTION PROCESSES

ABSTRACT

Stability of fibres becomes more important in mechanically-produced paper when exposed to changing environmental conditions such as moisture associated with production processes such as coating, supercalendering and printing. Mechanically-produced paper that is subjected to this calendering pressure, experience more movement during moisture as a result of pressed fibres that recover to their original shape more than uncalendered paper. Previous research investigated the impact of wood type, fibre morphology and enzyme treatments have on fibre stability during exposure to moisture. The investigation was based on fibre characterisation, handsheet properties and cross-sectional behaviour of mechanically-produced fibres in handsheets. The findings from previous studies reflected on laboratory produced calendered and rewetted handsheets and did not necessary reflect the behaviour of fibres in commercially produced paper sheets. The aim of the present study was to determine the stability of commercially produced paper from chemi-thermo-mechanical pulp across different production processes and also to compare the results obtained from laboratory-produced pulp and handsheets with commercial pulp and sheets. Surface and strength properties of commercial paper samples (base-sheet, coated, and supercalendered-paper) were tested. A significant increase in roughness was observed in all three samples after exposure to moisture and strength loss was most evident in supercalendered-paper after rewetting. These three samples (base-sheet, coated and super-calendered-paper) including a commercially printed paper sample was examined with a scanning electron microscope combined with image analysis. The puffing effect observed in the rewetted supercalendered paper was similar to that of a printed sample. This study on commercially produced paper across different production processes showed similar results to those obtained previously with handsheets after calendering and rewetting. Therefore, the reliability of test methods (calendering and rewetting) applied to handsheets on laboratory scale could be validated as a measure to quantify puffing and fibre stability.

6.1. INTRODUCTION

A key factor for high-quality printing is the stability of the fibres when exposed to changing environmental conditions such as moisture and heating. These changes in moisture occur during processes such as coating and printing. The moisture originates from water-based inks, ink-water emulsions or the aqueous fountain solution in offset printing (Biermann, 1996), whereas coating suspensions are also made up with water.

The response of fibres towards these changes in moisture, and the characteristics of the paper produced, are influenced by the fibre morphology and processing conditions. Previous research has shown that the fibre behaviour is influenced by the morphology of different wood species used in the furnish (Horn, 1974; Laine, 1997). Paper properties can also be controlled through processes such as blending, fractionation and refining of fibres, and coating and calendering of sheets (Biermann, 1996; Kure *et al*., 1999; Holmstad *et al*., 2004; El-Sharkawy *et al*., 2008). Coating is applied to improve the surface properties of the base-sheet in terms of smoothness, brightness, gloss and overall uniformity (Biermann, 1996). As a result, coatings also improve aspects important to printing process such as surface smoothness (Skowronski and Lepoutre, 1985). However, in some papers containing mechanical pulp, the interaction with water may lead to movement of unstable fibres as result of reduced inter-fibre bonding or fibre puffing (Aspler and Béland, 1994). Calendering or supercalendering is commonly applied after the paper surface is coated to remedy flaws such as high surface roughness. However, Skowronski (1990) reported that application of calendering pressure may introduce strain in the paper that can cause an even greater increase in roughness following exposure to moisture as unstable fibres tend to recover their original shape.

In order to understand the response of rewetted fibres, the behaviour of single-specie pulps (Strey *et al*., 2009; Chapter 5), as well as that of different fibre types (earlywood and latewood; Chapter 4) were investigated under controlled laboratory conditions. Fibre puffing was common in softwood pulp, contributing to surface roughness following rewetting (Strey *et al*., 2009).

In contrast, hardwood fibres such as poplar puffed relatively less after rewetting, maintaining both surface smoothness and strength properties (Chapter 5). The differences observed between the response of pulp from spruce and poplar were ascribed to corresponding differences in coarseness, collapsibility, as well as upon the amount of fibrillation, all of which contribute to fibre stability.

While the trends observed have increased present understanding of fibre stability, these results have been obtained using selected raw material and processes under laboratory conditions, and thus still need to be validated under industrial conditions where a heterogeneous furnish is the norm. The aim of the present study was, therefore, to investigate the stability of fibres from a mixed furnish contained in commercial paper.

6.2. MATERIALS AND METHODS

6.2.1. Sampling

Three commercially produced paper samples containing a mixture of poplar (30%) and spruce (70%) were received from a bleached-chemi-thermo mechanical pulp (BCTMP) and paper mill. Samples were collected during a single sampling session from a mill that produces a base-sheet that subsequently undergoes on-machine calendering, coating and off-machine supercalendering, depending in the end product required. The base-sheet sample (40 g m^{-2}) was collected after on-machine calendering. The second sample (80 g m^{-2}) was collected after coating, and a third was a supercalendered sample. These three samples were tested by comparing surface smoothness, tensile and burst strength before and after rewetting, as well as fibre puffing after rewetting. A fourth sample of the same product was a coated-supercalendered paper which had been printed on both sides by a commercial printer. This sample allowed the extent of puffing occurring under commercial printing conditions to be determined.

6.2.2. Properties of commercial paper samples

Surface roughness, tensile and burst strengths of the three commercial paper samples (base-sheet, coated-sheet and supercalendered-sheet) were measured using appropriate ISO methods as described in Strey *et al*. (2009). Corrected strength indices, based on a 40 g m^{-2}

base-sheet were calculated for each sample. Scanning electron microscopy (SEM) was used to examine the surface structure of these samples. As before, image analysis was use to determine the lumen area to fibre area ratio (LA:FA) of dry sheets and the impact of rewetting on the cross-sectional behaviour of fibres in the base-sheet, coated-paper, supercalendered-paper and a commercially-printed sample.

6.2.3. Experimental design and statistical analysis

Given the limited size of the samples provided, replicated evaluation of the material was not possible. Pairs of treatments were compared using the Student's t-test at a 95% confidence level. The properties compared were roughness, burst and tensile indices as well as puffing (LA:FA ratio) for dry and rewetted samples.

6.3. RESULTS AND DISCUSSION

6.3.1. Properties of commercial paper samples

Coating, followed by supercalendering increased the smoothness of base-sheets as observed in SEM micrographs (Fig. 6-1). However, the relative increase in smoothness was most evident in supercalendered-paper (Figs. 6-1 and 6-2). This effect was probably due to the smoothening effect of the calendering rollers on the coated surface (Fig. 6-1c) and the corresponding increase in compression of the fibres.

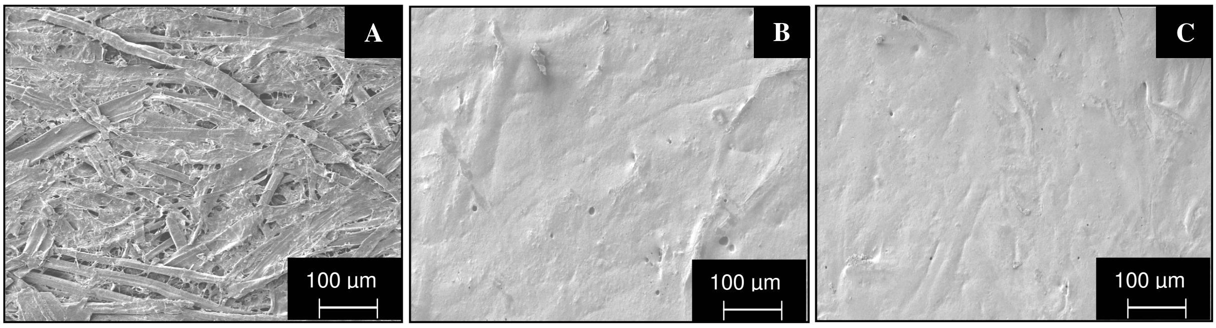

Figure 6 - 1: *Representative scanning electron micrographs showing the surface structure of the A: base-sheet, B: coated-paper and C: the supercalendered-paper.*

After rewetting, the surface roughness of all three paper samples increased significantly when compared to the dry samples (Fig. 6-2). The base-sheet showed only a two-fold increase in roughness after rewetting, compared to the five-fold in the coated-paper and a 42-fold increase in the supercalendered-paper. Notably, the roughness of the base-sheet from this mixed furnish increased more after rewetting than the corresponding changes observed in handsheets from spruce in a previous study (Strey *et al*., 2009). This large increase was possibly due to more effective calendering achieved in the mill than achieved during calendering of handsheets in the laboratory. Commercial calendering possibly led to greater compaction of fibre in the network and supports the conclusions of Skowronski (1990).

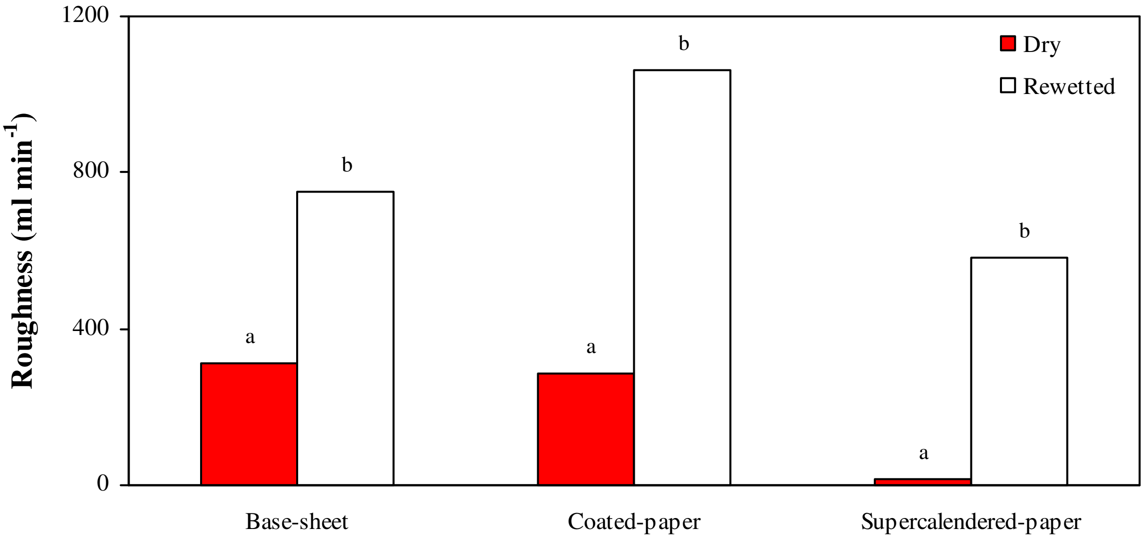

Figure 6 - 2: *The influence of rewetting on roughness of commercial paper from a commercial paper mill. (Columns for each type of paper with the same letter do not differ significantly).*

Coating contributed to an increase in strength properties when compared to the base-sheet (Figs 6-3 and 6-4), but tensile and burst strengths of the coated and supercalendered-papers were similar (Figs 6-3 and 6-4, respectively), suggesting that the additional calendering pressure had no additional effect on paper strength. Rewetting did not influence the tensile or burst strength of the base-sheet or coated-paper when compared to the dry samples (Figs 6-3 and 6-4), given that the calendering pressure of the base-sheet and coated-paper sample was the same. In contrast, both tensile and burst indices of supercalendered-paper samples were lowered by rewetting. It is possible that the higher pressure exerted on paper during supercalendering induced greater strain into the fibre network upon drying, and that during rewetting this strain was relieved, leading to movement of unstable fibres, lower inter-fibre bonding and loss of paper strength.

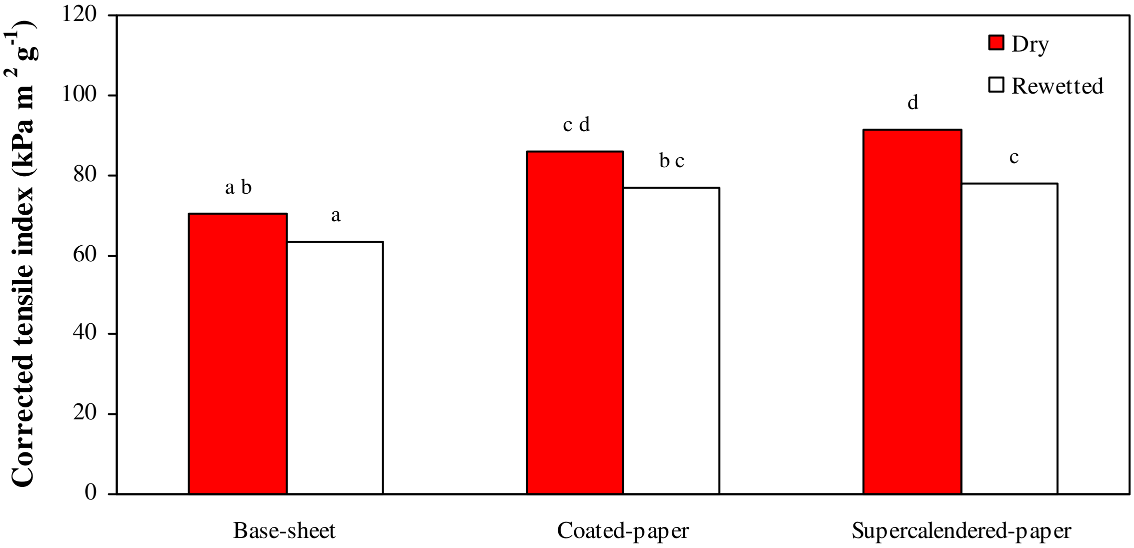

Figure 6 - 3: *The influence of rewetting on the corrected tensile index of production paper from a commercial paper mill. Columns with the same letter do not differ significantly, p≤0.05 (pairs of treatment means were compared using a Student's t-test).*

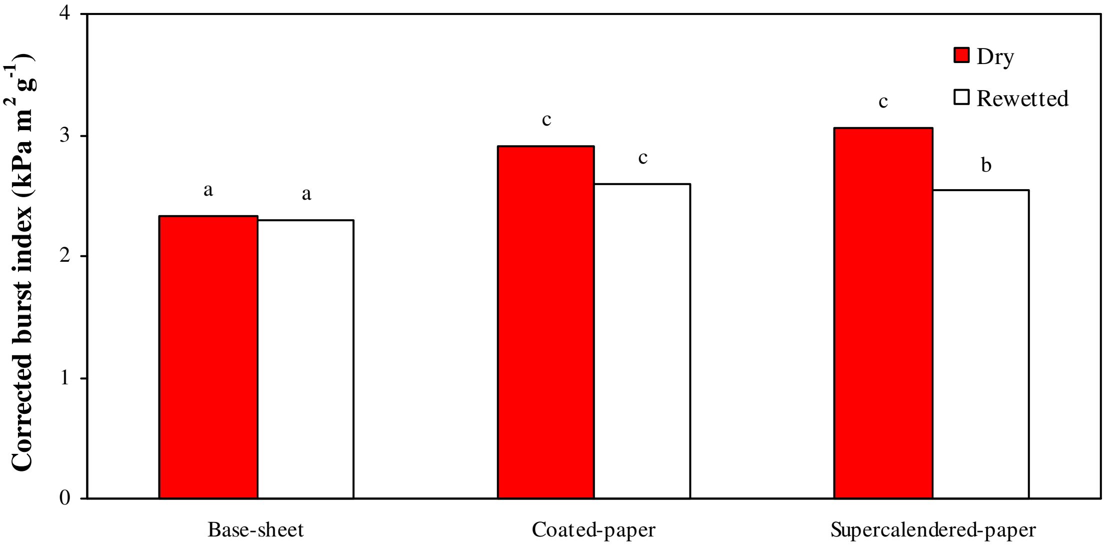

Figure 6 - 4: *The influence of rewetting on the corrected burst index of production paper from a commercial paper mill. Columns with the same letter do not differ significantly, p≤0.05 (pairs of treatment means were compared using a Student's t-test).*

6.3.2. Stability of fibres in commercial paper

Fibre instability is reflected in the degree of puffing defined earlier as the increase in the ratio of lumen area (LA) to fibre area (FA) (Strey *et al.*, 2009). The LA:FA ratio of the dry samples showed that the base-sheet contained a significantly larger amount of uncollapsed fibres compared to the more collapse fibres in the coated and supercalendered papers (Table 6-1; Fig. 6-5). Comparing the coated and supercalendered paper, no significant changes were observed in the LA:FA (p=0.44), although it was expected that the pressure applied to the fibres during supercalendering would lead to greater fibre collapse (Skowronski, 1990) and thus a significant reduction in LA:FA ratio.

Table 6 - 1: *Mean LA:FA ratio of dry and rewetted samples from different production stages and printed-paper.*

Commercial paper samples	Dry	Rewetted	Significance [a]
Base-sheet (LA:FA)	0.214	0.272	p = 0.38
Coated-paper (LA:FA)	0.188	0.316	p = 0.01
Supercalendered-paper (LA:FA)	0.167	0.252	p = 0.04
Printed-paper (LA:FA)	0.256	--	--

a: Values of p determined in a t-test comparing dry and rewetted treatments of the same sample.

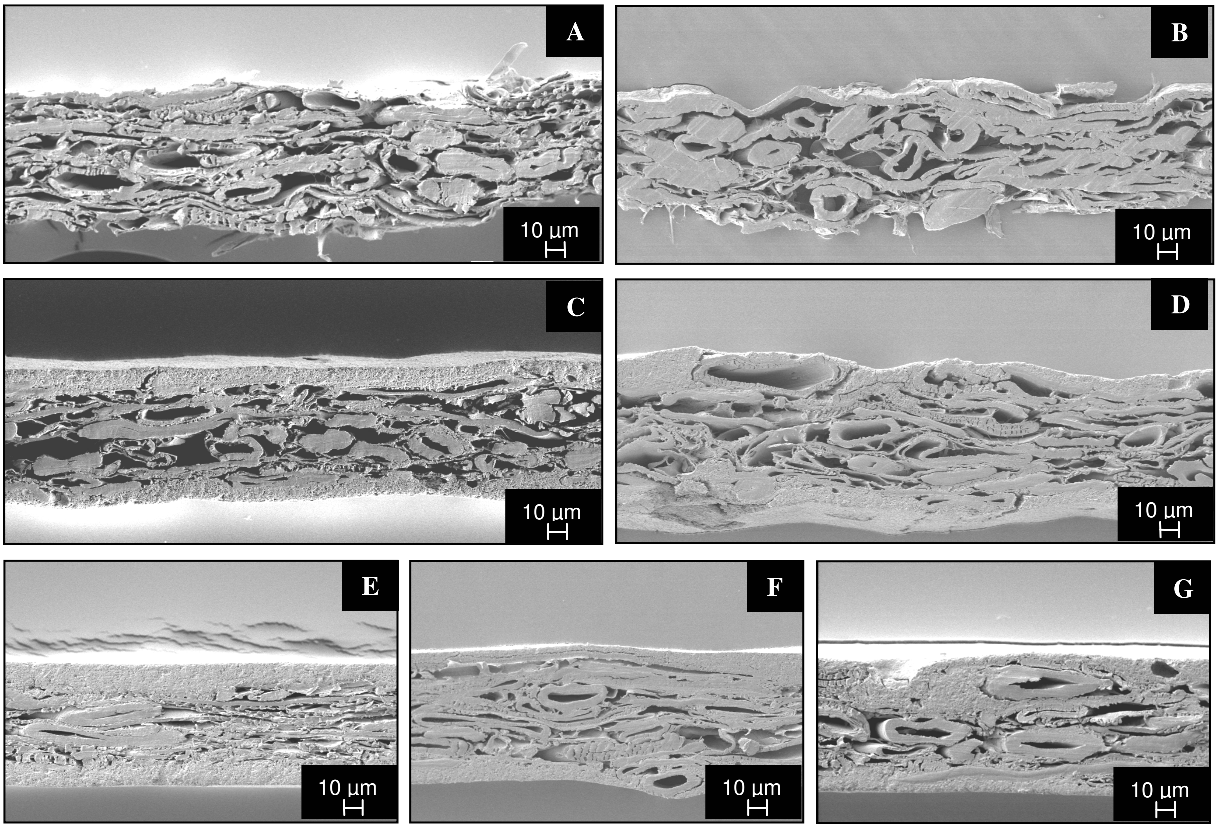

Figure 6 - 5: *Transverse sections of dry and rewetted samples from different treatments (A: dry base-sheet, B: rewetted base-sheet, C: dry coated-paper, D: rewetted coated-paper, E: dry supercalendered-paper, F: rewetted supercalendered-paper and G: printed-paper).*

Significant increases in the LA:FA ratio after rewetting were observed only in the coated and supercalendered samples, but not in the base-sheet (Table 6-1; Fig. 6-5). After rewetting an increase in puffing was observed in coated-paper but not in the base-sheet. It was unexpected because, as mentioned earlier, both the base-sheet and coated-paper were subjected to the same pressure. Therefore, it is possible that supercalendering pressure has an effect on fibre puffing (as was initially thought), but an interaction between fibres and the water-based coating suspension could also lead to puffing (Table 6-1). However, there is incomplete information in this study to support or reject this suggestion.

One of the critical questions addressed in this study was whether or not rewetting conducted under laboratory conditions replicated reliably the extent of rewetting that occurs during commercial printing. Results indicate that the LA:FA ratio of supercalendered-paper after printing (Fig. 6-5g) was significantly greater (p=004) than that of the dry supercalendered-paper (Fig. 6-5e; Table 6-1), indicating that puffing occurred. Furthermore, the LA:FA ratio of the supercalendered-paper after rewetting (Fig. 6-5f) was similar compared to the printed sample (0.252 and 0.256, respectively), indicating that the extent of puffing did not differ significantly (p=0.94). The rewetting of supercalendered-paper in the laboratory had a similar effect to that of the printing process.

6.4. CONCLUSIONS

Laboratory studies indicate that the response of paper to rewetting is influenced by the type of wood fibres present (Strey et al., 2009; Chapters 2; 4 to 5) and that the relative stability of fibres is influenced by processes such as calendering. However, it was important to test the validity of these laboratory findings against paper from mixed furnish produced under commercial processing conditions.

The paper properties of the commercial samples were measured before rewetting and surface smoothness was increased by supercalendering. Strength (corrected tensile and burst indices) was not influenced in the base-sheet or coated-paper after rewetting. However, after supercalendering, a reduction in both these strength properties was observed when fibres were exposed to water. Therefore, in the present study, when supercalendered-paper was rewetted the presence of unstable fibres was confirmed. Since the majority of the furnish consisted of spruce (70%) it was assumed that the long fibres were the source of instability. Previous work on spruce showed that rewetting of handsheets caused increased roughness while tensile and burst strength were reduced due to unstable fibres (Strey *et al*., 2009). In contrast, poplar fibres were more stable; therefore, surface and strength properties could be maintained after rewetting (Chapter 5).

Calendered fibres have a greater tendency to react with moisture and move, because of the strain introduced to the paper (Skowronski and Lepoutre, 1985). The present study showed that unstable fibres are more evident in paper with fibres under strain caused by supercalendering, compared to the base-sheet and coated-paper. The lack of change between the base-sheet and coated-paper after rewetting reflects more stable fibres. However, these samples (base-sheet, coated-sheet and supercalendered-paper) contained the same furnish, therefore, highlighting the important role of the converting processes in controlling fibre puffing.

The dry base-sheet contained the largest amount of partially-collapsed fibres, as shown by the largest LA:FA ratio observed in SEM images. After supercalendering was applied, the smallest LA:FA ratio was measured. Calendering increased fibre collapse in the present study, but during rewetting the instability of these fibres became evident. The puffing effect in the base-sheet was insignificant, but in the coated and supercalendered samples, significant puffing was observed reflecting the presence of unstable fibres.

For validation of laboratory calendering and rewetting processes, a printed sample was evaluated and compared to the dry and rewetted commercial samples. The magnitude of puffing observed in rewetting of supercalendered-paper samples was similar to the amount of puffing observed in the printed-paper sample of the same product. It was concluded that the technique of rewetting successfully imitated the effects of the printing process and the test method (calendering and rewetting) applied to handsheets was validated as a technique to measure puffing and quantify stability. However, care should be taken in interpreting these results, since the sampling was not replicated.

6.5. REFERENCES

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